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BENEFICIATION, CHARACTERIZATION AND APPLICATION OF AKPET 1 BARITE FOR POTENTIAL USE IN DRILLING OPERATIONS

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Abstract

Successful drilling relies on the properties of the drilling fluid used to drill the wells. Barite serves as a weighting agent in the production of drilling fluid. Over the years, Nigeria's oil and gas industry has relied largely on imported barite for drilling operations, while the country has vast reserves of barite. There is a need to evaluate the properties of locally sourced barites for their suitability for drilling fluid production. Total reliance on foreign-produced barites has been the trend because of the oil firms in the nation establishing that the locally made barites are of low quality in chemical and physical properties and the quality is below American Petroleum Institute and Nigeria's Department of Petroleum Resources barite quality standard. Therefore, this study evaluates the best physical and chemical processes to prepare locally made barites using Akpet 1 barite ore as a case study. The characterization and XRD results of the on-site barite reads 62.101% $BaSO_4$, 15.423% of SiO_2, 2.201% of Fe_2O_3, 5.213% of Al_2O_3 and other soluble salts, which after a series of beneficiation processes produced barite of 91.212% $BaSO_4$, 1.011% SiO_2, 0.414% Fe_2O_3, 0.751% of Al_2O_3. In addition, the specific gravity is 4.39, while the Mohr's hardness scale after beneficiation is 3.25 and the pH value is 6.8. These results and the percentage increase of $BaSO_4$ on Akpet 1 barite demonstrate that locally-made barites can be prepared to meet the international standard for drilling oil mud formulation. The number of impurities was reduced sufficiently low, and the specific gravity of the

samples improved to meet the needs of any drilling operation and compare favourably with industrially accepted barite

Keywords: Akpet 1 barite ore, Beneficiation, Characterization, Leeching, Specific gravity, weighting, Drilling Fluid.

INTRODUCTION

Petroleum is a mixture of a very large number of different hydrocarbons. The most commonly found molecules are alkanes (paraffins), cycloalkanes (naphthene), aromatic hydrocarbons, or more complicated chemicals like asphaltenes. These products are in two different phases which are gaseous phase (natural gases) and liquid phase (crude oil).

The contribution of crude oil to the growth of Nigerian economy has always been the paramount source of economic advantage to other neighbouring countries due to is a local production asset (Usoro *et al.*, 2020). An oil well is a boring in the Earth that is designed to bring petroleum oil hydrocarbons to the surface which is crude oil. Crude oil production is a product of the oil well after much drilling activities using drilling mud which aid the drilling of boreholes into the earth. However, due to uncontrollable technical pressures that result to explosion due to negative pressure during drilling, weighting substance like barite is introduced as additive into the drilling mud in order to keep safe environment (Afoloyan, *et al*., 2021).

The high specific gravity of barite makes it suitable for a wide range of industrial, medical, and manufacturing uses. Barite also serves as the principal ore of barium ($BaSO4$). Barite is also used as a pigment in paints and as a weighted filler for paper, cloth and rubber. It is the primary ore of barium, which is used to make a wide variety of barium compounds. Some of these are used for x-ray shielding. It has the ability to block x-ray and gamma-ray emissions. Barite compounds are also used in diagnostic medical tests. If a patient drinks a small cup of liquid that contains a barium powder in a milkshake consistency, the liquid will coat the patient's esophagus

However, the overwhelming majority of the barite that is mined is used by the petroleum industry as a weighting material in the formulation of drilling mud. Barite increases the hydrostatic pressure of the drilling mud allowing it to compensate for high-pressure zones experienced during drilling. The softness of the mineral also prevents it from damaging drilling tools during drilling and enables it to serve as a lubricant.
Nigeria is richly and abundantly blessed with solid minerals of various types such as iron ore, tantalite, barites, gold, tin, bentonite, gypsum etc (Duru, *et al*, 2019*).* The mining sector in Nigeria is only contributing about 0.5% – 0.6% to the country's GDP because of her dependence on oil resources. The mining industry in Nigeria is underdeveloped, resulting in the importation of minerals that could be produced domestically (Ebunu, *et al.,* 2021). There are about 44 different types of minerals that have been identified in over 500 locations in Nigeria and one of such minerals is barite. Barite occurs in veins, stratiform beds and residual deposits. Stratiform beds are the largest global deposits currently mined in the United States (US), China and India (Duru, *et al.,* 2019).
In Nigeria, locally sourced barites are mainly used in glass production, paints production and other industrial productions excluding crude oil drilling production due to the crude oil producing companies (mainly foreign firms) discouraging it for lack of purity percentage and quality. Total reliability on foreign barites for crude oil drilling by Nigerian Oil and Servicing companies without applying the use of locally made barites has not contributed enough impact to the nation’s Gross Domestic Product and internal revenue generation. The challenging issue has been that our barites production is not of international standard and need to be prepared to an acceptable global quality. (Ebunu, *et. al.,* 2021).
Crude oil drilling activities usually experience technical difficulties like wearing of drilling equipment, breaking down of drilling machines and build-up of negative pressures even with drilling mud if a good weighting additive like barite is not added to eliminate the negative impact (Usoro, *et al.,* 2020) However, the natural formation barite ore which has been discovered in Nigeria for more than fifteen years (15 years) now has not

been fully applied by both local and foreign oil production and servicing companies in their drilling and oil production activities.

Economic diversification aimed by Federal Government of Nigeria to enhance Gross Domestic Product has not fully enforced by the crude oil drilling companies with the use of domestic produced barites in their daily crude oil generation activities due to assumption of low quality and lack of trust on our domestic produced barites (Mgbemere, *et al*., 2018). On this account, beneficiation of Nigerian Barite ore is necessary to ascertain and ensure its quality meets up the imported ones in crude oil drilling activities by characterizing and improving its quality (Salahudeen, *et al*., 2020).

In addition, The Federal Government has placed a ban on further importation of barite from other countries but mainly encourage industrial users to buy locally made barites. This move is encouraged by terminating foreign exchange budget for it thereby saving $300 million every year in foreign exchange (FX). (Chukwu ,2021,28th October).

The lack of this appropriate information on the mineral stores keeps on comprising a significant obstruction to their financial usage. Surely, nearly all past research focused at Barium Sulphate ore mineralization in the Akpet 1 in Biase Local Government of Cross-Rivers State trough have, until recently, generally centered on the origin and discovery of the ore vein and uses in other industries excluding oil drilling.

On this vein, this research study is on the effective production of crude oil drilling mud using

Nigeria purified barite from Akpet 1 in Base Local Government of Cross-Rivers State in

Nigeria. The emphasis on the research is on beneficiation of our local barite ore using Akpet 1 deposit as case study to attain a standard specific gravity between 3.8 - 4.5 and 88 – 98% of Barium Sulphate (BaSO4) compound, which gives good weighting during crude oil mud formation.

MATERIALS AND METHOD

The materials are Akpet 1 Barite ore sourced from barite mine in Biase Local Government of Cross Rivers State., Oleic acid, Pine oil and Hydrochloric acid (HCL). These were purchased from Parsity Laboratory and Chemical Ltd and Cardinal Scientific Supplies, all are in Kaduna State, Nigeria. In addition, Ionized water was also prepared in the laboratory for beneficiation.

Method:

Sample Preparation:

The bulk samples collected were sampled uniformly for standard sampling to yield representative sample. In laboratory, the sample was divided into two (2) portions for initial physical testing and continuation of beneficiation processes. The second sample was washed with fresh water to remove any dust and other external matters trapped or they may be contained on their external surfaces. The washed samples were dried in an oven at a temperature of 50-degree Celsius (50°C) for a week. Dry barite bulk sample was crushed with clean hammer and then poured into a Jaw crushing machine to convert them to the required powder grade.

The crushed sample was introduced into Ball mill which was used to mill the sample into powder form, sieved and screened with sizes ranging 3 mm to 45 microns with the aid of vibratory screen. This was achieved with the aid of a mechanical sieve shaker/size analyzer which comprises an agitator coupled with a mesh of various sizes, ranging from 3 mm to 45 microns. The samples were poured gently into the upper sieve while lowering the samples across the various mesh designations, to collect the finest samples at the receiver plate.

Sieve in micrometer (µm)	Percentage of passing	Approved Standard
100	70	50 - 85
80	55	35 - 70
60	45	25 - 60
45	32	15 - 50
30	24	10 - 30
15	7	2 - 10

Therefore, the sample mixing is consisted of **100% of 15 µm, 85% of 30 µm, 70% of 45µm, 50% of 60µm, 30% of 80µm and 10% of 100µm.** This is to present a comprehensive representation of uniform sample with good surface area for beneficiation. However, the raw sample was taken for initial characterization using X-ray fluorescence (XRF), X-ray diffraction (XRD), initial flame test and initial specific gravity analyses. Also, Furrier Transform infrared (FTIR), Thermogravimetric analysis (TGA) and Scanning Electron Microscopy (SEM) tests were carried out on raw barite sample. This is to ascertain the physical and chemical properties of the sample and the content percentage of $BaSO_4$. Before beneficiation processes.

Jigging method Analysis Test:

1000g of the uniformed milled sample was poured on the jigging machine for gravitational separation. The particle sizes in the range from 100 to 15µm were used to produce the pulp of the ore for the jigging experiment. The pulp was fed into the jigging equipment with a constant supply of water. The overflow was collected into the collecting pan while the underflow was also collected and both were allowed to settle and dewatered. Both the underflow and the overflow were dried in an oven for about 5hours and their weights were measured. The resultant sample was characterized using XRF.

Froth Floatation Method Test:

250g of the underflow which is the concentrate from the jigging operation was subjected to froth flotation by mixing with 2000 ml of distilled water. This was done in a Denver flotation cell operating at an impeller speed of 1500 rpm for about 5 minutes to obtain proper suspension of solid particles within the pulp. Pine Oil, Oleic acid, and HCl is reagent that were used for the Froth Flotation process as frother, collector and pH regulators respectively. Four (4) drops of the oleic acid were added to the pulp and conditioned for about 5 minutes. Starch acting as a depressant was added and conditioning in vacuum was carried out for about 10 minutes. The pine oil was added 2 mins before the expiration of the conditioning time while allowing air to be introduced at a reasonable rate. The froth was skimmed off the flotation cell into the collecting pan until barren froth persisted. The froth was allowed to settle, dewatered by filtration, washed and dried in the laboratory oven at 200°C for 5 hours. The depressed mineral was also dewatered, washed and dried. This was done at pH value of between 6.0 – 7.0. The resultant sample was characterized using XRF.

Leaching Test using Acid Reagents:

Acid leaching test performed on final flotation concentrate using concentrations of Hydrochloric acid (32.36%). 25g portions of concentrate were taken in to Pyrex glass beakers (500 mL), treated with 100 mL of diluted acid (0.2M of HCL), with constant stirring on hot plate at 95°C to boiling temperature for 1 hour with speed of 10 rpm. The analysis was cooled, filtered and washed with distilled water until it is free of the acid. The residues left on filter paper were dried for 20 minutes in electric oven at 110 °C and the final product was examined using XRF and XRD machine for Barite content.

Characterization of Acid Leeched Akpet 1 Barite:

The beneficiated Akpet1 barite was characterized to determined its properties and percentage content of BaSO4. This was done using X-ray fluorescence, X-ray diffraction (XRD), flame test, specific gravity bottle, and other physical test like hardness, Loss on Ignition, moisture content, pH. The Furrier Transform infrared (FTIR) was used to the ascertain the bioactive functional groups present in the and compared to the leeched barite and compared to the raw barite, while Scanning Electron Microscopy (SEM) helped to study the morphology of the both raw and beneficiated barites.

RESULT AND DISCUSSION

Characterization of Raw Akpet 1 barite ore:

Table 1: Characterization result of Akpet 1 barite before beneficiation

Major Components	SiO_2	Fe_2O_3	P2O5	SO_3	CaO	K_2O	BaO	Al_2O_3	TiO_2
Concentration %	**15.423**	**2.201**	**6.708**	**13.016**	**2.843**	**2.891**	**49.085**	**5.213**	**0.824**

From the XRF analysis result above, Silica oxide (SiO_2) has more percentage on the barite which is a major formed contaminant, together with Alumina (Al_2O_3) and other oxides. These are major components of clay, laterite and some earth solid deposits. However, barite international standard has to have 84 – 98% $BaSO_4$. The chemical equation of the analysis is

$BaO + SO_3 \rightarrow BaSO_4$ …………………. Eq 1

The percentage is; **49.085 + 13.016 = 62.101%**

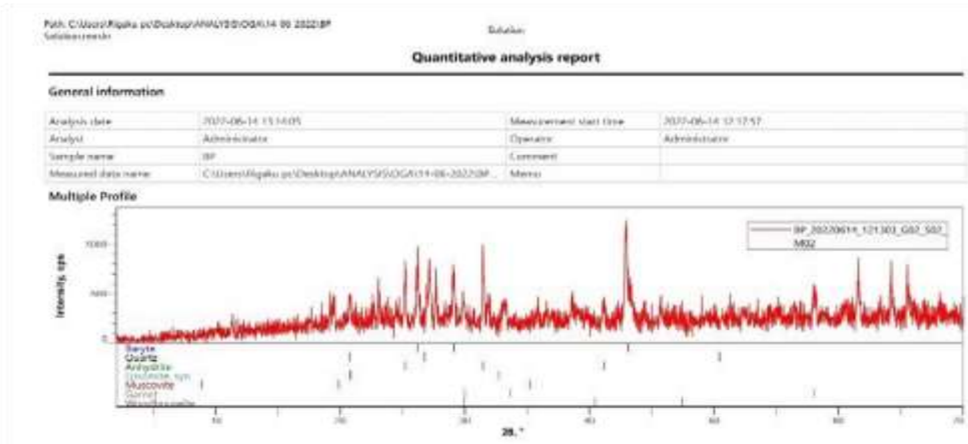
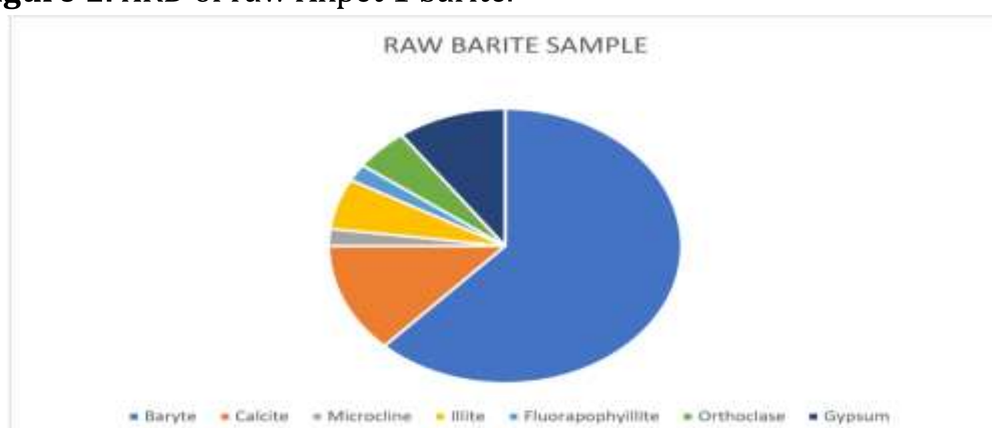


Figure 1: XRD of raw Akpet 1 barite.



Dataset / Weight Fraction, wt.%	Value, Unit	Baryte	Calcite	Micro cline	Illite	Fluora pophyl lite-(K)	Ortho clase	Gypsum
BARITE_20230207_144856_G04 ...	0	62(6)	13(18)	2(9)	6(6)	2(5)	5(4)	10(5)

Figure 2: Chart representation of the XRD result on Raw Akpet 1 barite ore.

The XRD shows a larger percentage of BaSO4 in the sample phases which establishes the study that with beneficiation (removing much percentages of other unneeded oxides) that better purified local barite will establish a global standard local barite for drilling oil usage.

Characterization of the Akpet 1 barite after Jigging Method Separation:

The overflow that was collected into the collecting pan was weighed after drying. M_1 = 295g (Overflow mass after drying); M_2 = 623g (Underflow mass after drying). 250g of the underflow which has high percentage of the barite is used in froth floatation process of purification. Moreover, 20grams of the sample of underflow was taken for characterization to determine the effect in percentage the process have on the Akpet 1 barite using XRF machine.

Table 2: Characterization result of Akpet 1 barite after Jigging Method of purification

Major Components	SiO_2	Fe_2O_3	P2O5	SO_3	CaO	K_2O	BaO	Al_2O_3	TiO_2
Concentration %	11.230	2.050	4.180	13.957	1.999	1.008	60.15	3.410	0.719

The percentage of **$BaSO_4$** is; **13.957 + 60.150 = 74.117%**

Froth Floatation Method of Purification.

This process was applied using Pine oil (mixture of chemicals), Oleic acid ($C_{18}H_{34}O_2$), Hydrochloric acid (HCL) and Sodium Hydroxide (NaOH) as reagents. The dried concentrate was measured. **M_3 = 125g** (barite concentrate after froth floatation process). 20grams of the concentrate was characterized to view the effect of the process on the sample using XRF and the result is shown below;

Table 3: Characterization result of Akpet 1 barite after Froth floatation process.

Major Components	SiO_2	Fe_2O_3	P2O5	SO_3	CaO	K_2O	BaO	Al_2O_3	TiO_2
Concentration %	**5.016**	**0.958**	**4.222**	**14.323**	**0.911**	**0.982**	**70.910**	**1.085**	**0.589**

The percentage of **$BaSO_4$** is; **70.910 + 14.323 = 85.233%**

Therefore, the need for continuing beneficiation using leeching method is necessary to be able to have a good weighting material for crude oil drilling application.

Leeching method with Hydrochloric acid:

Using Hydrochloric acid (HCL) to leech the concentrate was carried out and the result is shown below;

Table 4: Characterization of Leeched concentrate with HCL

Major Components	SiO_2	Fe_2O_3	P2O5	SO_3	CaO	K_2O	BaO	Al_2O_3	TiO_2
Concentration %	**1.011**	**0.414**	**4.300**	**15.418**	**0.512**	**0.256**	**75.794**	**0.751**	**0.337**

The percentage of **$BaSO_4$** is **74.794 + 15.418 = 91.212%**

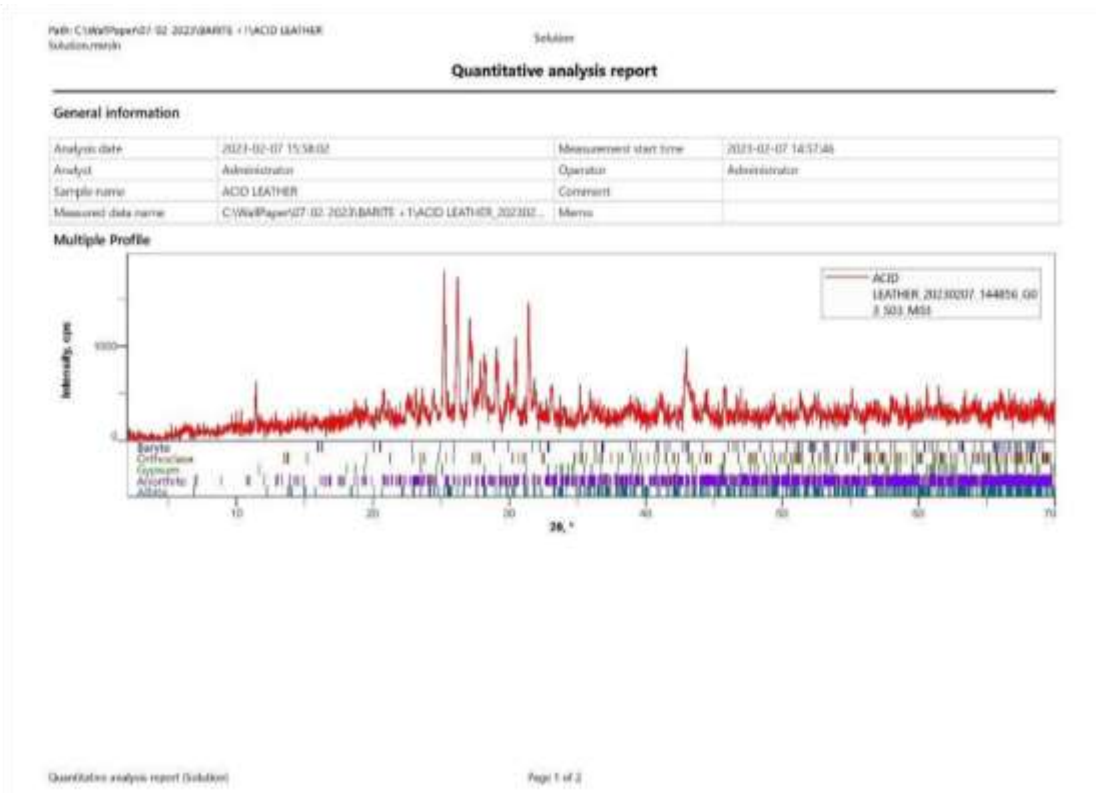
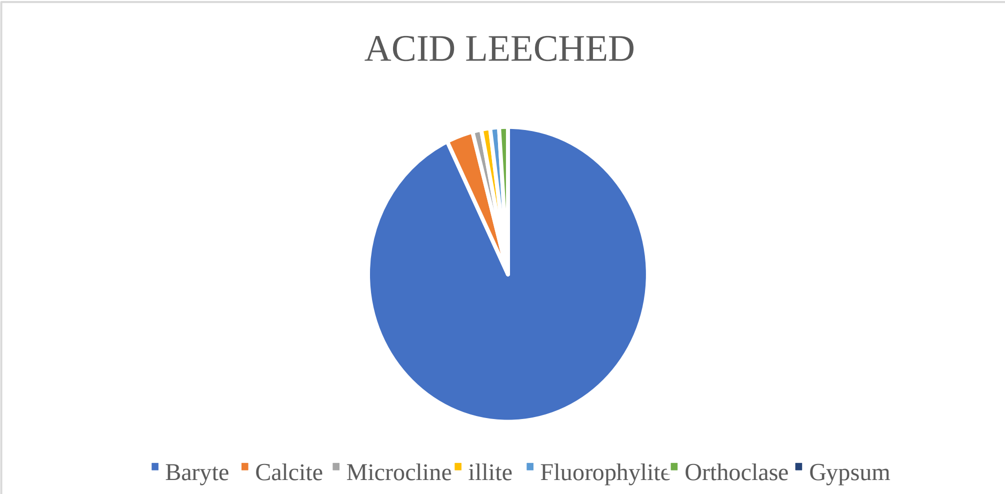


Figure 3: XRD representation of Acid leeched Akept 1 barite.



Orthoclas Gypsu e m 1(4) 2(5)

Dataset / Weight Fraction, wt%	Unit	Baryte	Calcite	Microcline	Illite	Fluorapophyllite-(K)
BARITE_20230207_144856_G04...	0	91(6)	3(18)	1(9)	1(6)	1(5)

Figure 4: Chart representation of the XRD result on Acid Leeched Akpet 1 barite ore.

The projected phases are phases with $BaSO_4$ which entails greater percentage of barium.

Flame Test on the sample:

The flame test before beneficiation was carried out and the picture below reflects a different colour from barite flame test. This shows that the raw barite ore has impurities and some external matters which affect the colour of its flame test. The flame colour is yellowish (Sodium compound suspected), gold yellow (Charcoal powder suspected) and Orange (Calcium compound suspected). The flame test done on the Akpet 1 beneficiated barite above picture shows the yellowish green the pure barite and depicts

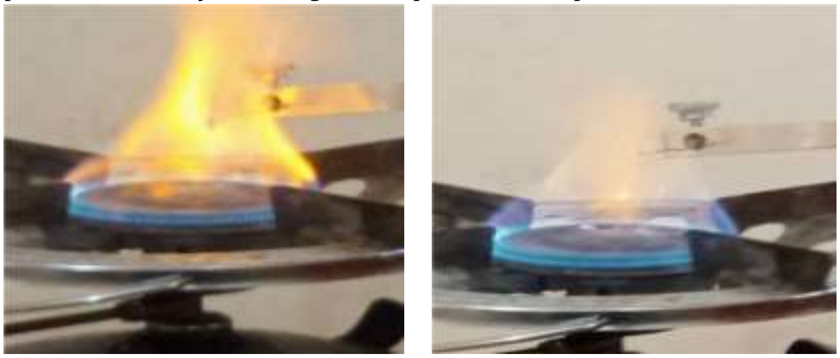


Figure 5: Initial flame test on the raw sample and flame test of beneficiated barite ore

Specific Gravity Test Results:

The tests were carried out before beneficiation and after beneficiation. The reason is to determine the specific gravity of the Akpet 1 barite ore when not purified to proof its percentage purity. The International Standard for specific gravity range of Barite is 4.2 – 4.6. The initial specific gravity taken using pycnometer bottle (density bottle) is shown below and average specific gravity measured.

$$\text{Specific gravity of soil} = \frac{\text{Density of water at } 27°C}{\text{Weight of water of equal volume}}$$

$$= \frac{(W2 - W1)}{(W4 - W1) - (W3 - W2)} \quad \text{........................ Eq2}$$

Table 5: Initial Specific Gravity of Akpet 1 Barite before Beneficiation

Parameters	1	2	3
Wt of empty density bottle (W_1)	23.30	23.30	23.30
Wt of empty density bottle + sample (W_2)	8.30	28.30	28.30
Wt of empty density bottle + sample + water (W_3)	74.95	74.97	74.93
Wt of density bottle + water (W_4)	71.60	71.60	71.60
Specific Gravity	**3.03**	**3.06**	**2.99**

Average. Specific Gravity before beneficiation is 3.03

Table 6: Specific Gravity of Akpet 1 Barite after Beneficiation

Parameters	1	2	3
Wt of empty density bottle (W1)	23.30	23.3	23.3
Wt of empty density bottle + sample (W2)	27.90	27.90	27.90
Wt of empty density bottle + sample + water (W3)	75.20	75.15	75.10
Wt of density bottle + water (W_4)	71.60	71.60	71.60
Specific Gravity	**4.60**	**4.38**	**4.18**

Average. Specific Gravity after beneficiation is 4.39

Moisture Content Result

$$\text{Moisture \%} = \frac{\text{loss in weight due to removal of moisture}}{\text{weight of baryte sample taken}} \, x100 \quad \text{Eq 3}$$

= ((initial weight – final weight after drying)/initial weight) * 100

Initial weight of the sample, S_1 = 5.00g
Final weight of the sample, S_2 = 4.91g
Moisture Content = ((5 – 4.91)/5)) * 100 = 1.8%

Relative Hardness and pH value results:

The relative hardness of the leeched Akpet 1 barite sample was determined using Mohs's hardness scale of table and its comparative procedure. Calcite, Gypsum, pocket knife and fingernail were used to scratch the Akpet 1 barite. The Barite was found to have relative hardness of 3.0-3.5 with average of 3.25 on the scale.

Also, pH value of the barite was found to be between 6.0 – 7.6 and average of 6.8, that is neutral.

Table 7: Relative Hardness and pH value

Property	**Feature**
Hardness of sample	**3.25**
pH	**6.8**

The FTIR results both raw and beneficiated Akpet 1 barite:

The result of raw sample shows inconsistency on the bands which explain the presence of more oxides in the sample while the later shows less vibrations graph at the same wavelength.

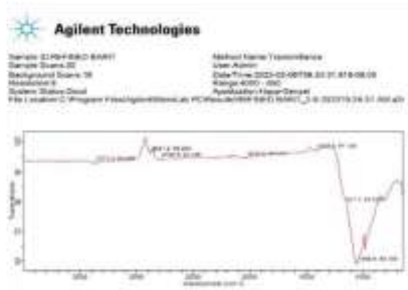


Table 8: Comparison with Barite API standard for physical and chemical requirements

Requirement	Numerical Value	Akpet 1 Beneficiated barite
Specific gravity	3.8 – 4.5 (4.2 minimum)	4.39
Moisture	1% - 2% maximum	1.8%
Flame colour	Yellowish green, Green lightened	Yellowish green
pH	7% maximum	6.8
Morh's Hardness	3.0 – 3.5	3.25
Barium Sulphate	88 – 95%	91%
Colour	Plain White, milky white	white

CONCLUSION

The concluded study has demonstrated that Akpet 1 barite ore can be purified to attain to the international standard, especially American Petroleum Institute physical and chemical standard. This supports the trend the Federal Government of Nigeria has taken to ensure total ban on importation of foreign barite which has not contributed to the gross domestic product earnings to the nation.

The building up $BaSO_4$ percentage was the major challenge having because some of local deposits are impure with more oxides the processes of purification used enabled the achievement of the target. Having 91% content of $BaSO_4$ after beneficiation encourages the Oil servicing company to follow the government order and invest in the local content.

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REFERENCES

Afolayan, D., O, Adetunji, A., R., Onwualu, A., P., Ogolo, O. & Amankwah, R., K. (2021). Characterization of Barite Reserves in Nigeria for use as Weighting Agent in Drilling Fluid. *Journal of Petroleum Exploration and Production Technology*. 11:2157–2178. Retrieved from doi.org/10.1007/s13202-021-01164-8

Chukwu, I. (2021,28th October). No Foreign Exchange for Barite as Federal Government to Ban. *Businessday Newspaper*. Retrieved from https://businessday.ng/news/article/no- fx-for-barite-as-fg-to-ban-import/.

Duru, U., I., Kerunwa, A., Omeokwe, I, Uwaezuoke, N. & Obah, B. (2019). Suitability of Some Nigerian Barites in Drilling Fluid Formulations. *Science Publishing Group*. 3(2): 46-59. Retrieved from doi: 10.11648/j.pse.20190302.13

Ebunu, A., I., Olanrewaju, Y., A., Ogolo O., Adentuji, A., R., & Onwualu, A., P. (2021). Barite as An Industrial Mineral in Nigeria: Occurrence, Utilization, Challenges and Future

Prospects. *Heliyon Journal and Books.* 7(6). Retrieved from doi.org/10.1016/j.heliyon.2021.e07365.

Mgbemere. H., E., Obidiegwu, E. & Obareki, E. (2018). Beneficiation of Azara Barite ore using a combination of Jigging, Froth flotation and Leaching. *Nigeria Journal of Technology*. 37(4): 957-962. Retrieved from dx.doi.org/10.4314/njt.v37i4.14.

Salahudeen, N., Yahya, M.N. & Mohammed, U. (2020). Morphological, Mineralogical and Chemical Characterizations of Azara Barite and its Yield Property as a Weighting Agent in Drilling Fluid. *Nigerian Research Journal of Engineering and Environmental Services.*5(2) 918-928.

Usoro, A. E., Ikpang, I. N., & George, E. U. (2020). Volatility measure of Nigeria crude oil production as a tool to investigate production variability. *African Journal of Mathematics and Computer Science Research*. 13(1), 1-16.